

JC07 Rec'd PCT/PTO 14 FEB 2002

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|---|--|---|--|---|--|
| FORM PTO-1390 (REV 11-98) | | U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE | | ATTORNEY'S DOCKET NUMBER | |
| TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371 | | | | 24230PCT/US | |
| | | | | U.S. APPLICATION NO. (If known, see 37 CFR 1.5) | |
| INTERNATIONAL APPLICATION NO. PCT/DE00/02890 | | INTERNATIONAL FILING DATE August 24, 2000 | | PRIORITY DATE CLAIMED August 24, 1999 | |
| TITLE OF INVENTION METHOD AND DEVICE FOR DEPOSITING MATERIALS WITH A WIDE ELECTRONIC BAND GAP AND A HIGH BINDING ENERGY | | | | | |
| APPLICANT(S) FOR DO/EO/US Johannes Kaeppler, et al | | | | | |
| Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information: | | | | | |
| 1. <input checked="" type="checkbox"/> This is a FIRST submission of items concerning a filing under 35 U.S.C. 371. | | | | | |
| 2. <input type="checkbox"/> This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. 371. | | | | | |
| 3. <input checked="" type="checkbox"/> This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1)(PCT/IPEA/401) | | | | | |
| 4. <input checked="" type="checkbox"/> A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date. | | | | | |
| 5. <input checked="" type="checkbox"/> A copy of the International Application as filed (35 U.S.C. 371(c)(2)) | | | | | |
| a. <input type="checkbox"/> is transmitted herewith (required only if not transmitted by the International Bureau). | | | | | |
| b. <input checked="" type="checkbox"/> has been transmitted by the International Bureau. (PCT/IB/308) | | | | | |
| c. <input type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US). | | | | | |
| 6. <input type="checkbox"/> A translation of the International Application into English (35 U.S.C. 371(c)(2)). | | | | | |
| 7. <input type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3)) | | | | | |
| a. <input type="checkbox"/> are transmitted herewith (required only if not transmitted by the International Bureau). | | | | | |
| b. <input type="checkbox"/> have been transmitted by the International Bureau. | | | | | |
| c. <input type="checkbox"/> have not been made; however, the time limit for making such amendments has NOT expired. | | | | | |
| d. <input type="checkbox"/> have not been made and will not be made. | | | | | |
| 8. <input type="checkbox"/> A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)). | | | | | |
| 9. <input checked="" type="checkbox"/> An ^{unsigned} oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)). (3 pages) | | | | | |
| 10. <input type="checkbox"/> A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)). | | | | | |
| Items 11. to 16. below concern document(s) or information included: | | | | | |
| 11. <input checked="" type="checkbox"/> An Information Disclosure Statement under 37 CFR 1.97 and 1.98. (2 pages of PTO-1449; 3 pages, one in English of EPO International Search Report listing references and their relevance; copies of 2 references discussed in specification and both in English) | | | | | |
| 12. <input type="checkbox"/> An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included. | | | | | |
| 13. <input type="checkbox"/> A FIRST preliminary amendment. | | | | | |
| <input type="checkbox"/> A SECOND or SUBSEQUENT preliminary amendment. | | | | | |
| 14. <input checked="" type="checkbox"/> Annexes to the specification ANNEXES, IF ANY, ARE NOT TO BE ENTERED | | | | | |
| 15. <input type="checkbox"/> A change of power of attorney and/or address letter. | | | | | |
| 16. <input checked="" type="checkbox"/> Other items or information: WO 01/14619 (with abstract) - cover sheet | | | | | |
| 17. <input checked="" type="checkbox"/> PCT/IPEA/401 | | | | | |
| 18. <input checked="" type="checkbox"/> PCT/IB/308 | | | | | |
| 19. <input checked="" type="checkbox"/> CLAIM IS HEREBY MADE OF THE BENEFIT OF THE FILING DATE OF German Patent Application 199 40 033.4 filed August 24, 1999 UNDER 35 USC 119. | | | | | |
| 20. <input checked="" type="checkbox"/> Express Mail mailing label No. EF024293082US deposited February 14, 2002 | | | | | |

JC13 Rec'd PCT/PTO 14 FEB 2002

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|---|-------------------------------|--------------------------|
| U.S. APPLICATION NO. (if known, see 37 CFR 1.5) | INTERNATIONAL APPLICATION NO. | ATTORNEY'S DOCKET NUMBER |
| 10/069031 | PCT/DE00/02890 | 24230PCT/US |

17. ☒ The following fees are submitted:

BASIC INTERNATIONAL FEE (37 CFR 1.492(a)(1)-(5)) :

| | |
|---|-----------------|
| Nor international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO | \$970.00 |
| International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO..... | \$870.00 |
| International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO | \$760.00 |
| International preliminary examination fee paid to USPTO (37 CFR 1.482) but all claims did not satisfy provisions of PCT Article 33(1)-(4).... | \$670.00 |
| International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(1)-(4) | \$96.00 |

ENTER APPROPRIATE BASIC FEE AMOUNT =

| | | |
|---|----|--|
| Surcharge of \$130.00 for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(e)). | \$ | |
|---|----|--|

| CLAIMS | NUMBER FILED | NUMBER EXTRA | RATE |
|--|--------------|--------------|------------------|
| Total claims | - 20 = | 2 | X \$18.00 |
| Independent claims | 2 - 3 = | 0 | X \$78.00 |
| MULTIPLE DEPENDENT CLAIM(S) (if applicable) | | | + \$260.00 |
| TOTAL OF ABOVE CALCULATIONS = | | | \$ 926.00 |
| Reduction of 1/2 for filing by small entity, if applicable. A Small Entity Statement must also be filed (Note 37 CFR 1.9, 1.27, 1.28). | | | \$ |
| SUBTOTAL = | | | \$ 926.00 |
| Processing fee of \$130.00 for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(f)). | | | \$ + |
| TOTAL NATIONAL FEE = | | | \$ 926.00 |
| Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property | | | \$ + |
| TOTAL FEES ENCLOSED = | | | \$ 926.00 |
| | | | Amount to be: |
| | | | refunded |
| | | | charged |

a. ☒ A check/in the amount of \$926.00 to cover the above fees is enclosed.

b. ☐ Please charge my Deposit Account No. _____ in the amount of \$_____ to cover the above fees.
A duplicate copy of this sheet is enclosed.

c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 06-0105. A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

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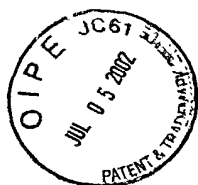
SIGNATURE

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NAME

Reg. No. 22,345

REGISTRATION NUMBER



10055033.1 10/05/02

#4.

23230PCT/US

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

USA PCT National Stage Patent Application
PCT/DE00/02890 filed August 24, 2000

Johannes Kaeppler, et al

Serial No.: 10/069,031

First Submission filed: February 14, 2002

METHOD AND DEVICE FOR DEPOSITING
MATERIALS WITH A WIDE ELECTRONIC
BAND GAP AND A HIGH BINDING ENERGY

CERTIFICATE OF MAILING ON LAST PAGE

Hon. Commissioner of Patents and Trademarks

Washington, D.C. 20231

S I R :

PRELIMINARY AMENDMENT

Please amend the translation of this application simultaneously
with filing the accompanying translation for this National Stage
application as follows:

IN THE ABSTRACT

An Abstract is presented herewith on a separate page.

IN THE SPECIFICATION

Page 1, Line 6, delete this line paragraph, namely "DESCRIPTION"

IN THE CLAIMS

Before claim 1, change "PATENT CLAIMS" to --WE CLAIM--

Please cancel claims 1-39 without prejudice or disclaimer of the subject matter therein and substitute claims 40-66 therefor:

--40. (new) A method for the deposition of semiconductor layers comprising SiC and/or $\text{SiC}_x\text{Ge}_{1-x}$ ($x=0 - 1$), AlN, GaN or related materials by a CVD method, wherein:

at least one substrate is heated to a temperature of approximately 1100°C to approximately 1800°C;

the at least one substrate rotates in an actively heated flow channel reactor;

the coating takes place homoepitaxially or heteroepitaxially;

at least one process or carrier gases is introduced just ahead of the hot substrate;

the flow channel reactor is heated on all sides; and wherein

the one or more process or carrier gases, before being introduced, are actively cooled to a temperature which is well below process temperature, so that premature decomposition of process gases and/or local supersaturation of gas stream with a decomposition product is avoided.

41. (new) The method according to claim 40, wherein the at least one substrate is disposed on at least one substrate holder plate, which is disposed in or on a substrate holder, and the at least substrate holder plate is driven relative to the substrate holder by "gas foil rotation".

42. (new) The method according to claim 40, wherein silane (SiH_4) or other Si-containing inorganic and organic starting materials, germane (GeH_4) and propane (C_3H_8) or other hydrocarbon gases are used as the process gas(es).

43. (new) The method according to claim 41, wherein by complete decomposition of source gases ahead of or above the at least one substrate, on account of a homogeneous temperature profile of the substrate holder, so that growth rates of $10 \mu\text{m/h}$ or more are achieved for the SiC and/or $\text{SiC}_x\text{Ge}_{1-x}$ ($x=0-1$) semiconductor layers.

a heater device which heats the substrate holder and thereby surfaces of the substrate holder which are to be coated, in a controlled manner to temperatures of from 1100°C to 1800°C; and wherein

a wall region of the reactor chamber which lies opposite the substrate surfaces which are to be coated are actively heatable to high temperatures; and

the gas inlet is coolable to a temperature which is well below process temperature.

47. (new) The device according to claim 46, wherein the reactor chamber is constructed in rotationally symmetrical form and has a central gas inlet and a rotationally symmetrical gas outlet.

48. (new) The device according to claim 46, wherein boundary walls of the reactor chamber which face reactor space, and at least one substrate plate and/or at least one said substrate holder have a continuous, inert coating, particularly comprising TaC, NbC, and the like, which is able to withstand high temperatures of up to 1800°C and cannot be etched by hydrogen radicals.

49. (new) The device according to claim 46, further comprising a turning device for rotation of the at least one substrate in each case on a substrate plate which is disposed in or on a substrate holder, by means of "gas foil rotation".

50. (new) The device according to claim 46, further comprising a turning device for rotation of the at least one substrate in each case on a substrate plate, which is disposed in or on a substrate holder, by means of a mechanically driven shaft.

51. (new) The device according to claim 46, further comprising at least one temperature control device for providing a uniform or different temperature to all boundary walls facing process gas, as top side, underside and side walls of a heated flow channel which is thereby closed off.

52. (new) The device according to claim 46, further comprising a combination of high-frequency, lamp and resistance heating means for heating the boundary walls which face process gas, and particularly the substrate holder.

53. (new) The device according to claim 51, wherein separate control of temperature of a substrate-side

boundary wall from an opposite boundary wall of the heated flow channel is effected by two separate heating circuits, each with a dedicated control means.

54. (new) The device according to claim 51, wherein boundary walls, which face the process gas, of the heated flow channel, and particularly a substrate plate and/or the substrate holder, are made from a highly conductive material.

55. (new) The device according to claim 51, wherein boundary walls, which face the process gas, of the heated flow channel, and particularly the substrate plate and/or the substrate holder, has a continuous, inert coating which is able to withstand high temperatures up to approximately 1800°C and cannot be etched by hydrogen radicals.

56. (new) The device according to claim 46, further comprising a cooling device actively cools the gas inlet, up to just before a heated flow channel, with a liquid or gaseous medium.

57. (new) The device according to claim 56, wherein the cool gas inlet is sealed with respect to the flow channel which is heated on all sides by means of a highly insulating, narrow adapter piece.

58. (new) The device according to claim 46, wherein a flow channel, downstream of an actively heated zone, comprises outlet segments which have different inert materials.

59. (new) The device according to claim 46, further comprising thin plates, compared to thickness of the substrate holder, of inert materials with a different electrical conductivity from the substrate holder are fitable on or in the substrate holder, in order to locally influence introduction of high frequency and thereby input of energy.

60. (new) The device according to claim 54, wherein the boundary wall, which lies opposite the at least one substrate, of the heated flow channel is installed in a fixed position, at a defined distance from a substrate-side boundary of the heated flow channel, or is rotatably connected thereto.

61. (new) The device according to claim 46, wherein a boundary wall, which lies opposite the at least one substrate, of a heated flow channel is actively coolable by a gaseous medium.

62. (new) The device according to claim 46, wherein there are a plurality of said at least one substrate which are disposed horizontally adjacent each other.

63. (new) The device according to claim 54, wherein the conductive material is graphite.

64. (new) The device according to claim 55, wherein said inert coating is TaC, NbC and the like.

65. (new) The device according to claim 58, wherein the different inert materials are TaC-coated graphite, SiC-coated graphite, quartz and the like.

66. (new) The device according to claim 59, wherein the inert materials are Ta, Mo, W.--

R E M A R K S

Claims 1-39 have been cancelled without prejudice or disclaimer of the subject matter therein and claims 40-66 respectively are presented to present said claims in accordance with USA practice under 35 USC 112, and to eliminate multiple-dependent form claims.


No multiple-dependent claim fees should apply in this application.

The specification has been amended for formal improvement to comply with USA practice.

An Abstract is presented on a separate page.

The Examiner is respectfully requested to enter this Preliminary Amendment prior to calculation of the filing fee as of the national stage filing date, and to provide an action on the merits.

Respectfully submitted
Johannes Kaeppler, et al

by: 
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USA PCT National Stage Patent Application
PCT/DE00/02890 filed August 24, 2000
Johannes Kaeppler, et al
Serial No.: 10/069,031
First Submission filed: February 14, 2002
METHOD AND DEVICE FOR DEPOSITING
MATERIALS WITH A WIDE ELECTRONIC
BAND GAP AND A HIGH BINDING ENERGY

ABSTRACT

A method and device for the deposition of semiconductor layers comprising SiC and/or $\text{SiC}_x\text{Ge}_{1-x}$ ($x=0 - 1$), AlN, GaN or related materials by a CVD method, wherein at least one substrate is heated to a temperature of approximately 1100°C to approximately 1800°C, the at least one substrate rotates in an actively heated flow channel reactor, the coating takes place homoepitaxially or heteroepitaxially, one or more process or carrier gases are introduced just ahead of the hot substrate. The flow channel reactor is heated on all sides, and wherein the one or more process or carrier gases, before being introduced, are actively cooled to a temperature which is well below process temperature, so that premature decomposition of process gases and/or local supersaturation of gas stream with a decomposition product is avoided.

உள்ளே சென்று மூன்று நாட்கள்

Method and device for depositing materials with
a wide electronic band gap and a
high binding energy

5

DESCRIPTION**Technical Field**

10 The invention relates to a method and a device for
depositing SiC and/or SiC_xGe_{1-x} (x=0-1) semiconductor
layers and related materials with a wide electronic
band gap and a high binding energy, such as for example
AlN or GaN, from the vapor phase and in particular by
15 means of a CVD method.

Prior Art

On account of their physical properties, semiconductors
20 with a wide band gap are particularly suitable for
applications which go beyond the range of use for
electronic semiconductor components based on Si or
GaAs. Chemical vapor-phase epitaxy (CVD) is the most
suitable method for producing electrically active
25 layers, such as SiC and/or SiC_xGe_{1-x} (x=0-1), for
electronic components for high-temperature, high-
frequency and high-power applications.

In the case of vertical, space charge zone-controlled
30 components, such as for example Schottky diodes or pn
diodes, for typical power applications it is necessary
to incorporate blocking voltages in the range $U > 10$ kV. It is therefore necessary for the epitaxial
layers which are deposited to have thicknesses of up to
35 100 μm .

The ability to switch high currents of $I > 10$ A also
involves high powers. Only large-area components are

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able to carry these high currents in the transmission region. The resulting constantly increasing demand for substrates with a minimum diameter of 4'' (inches) requires, for epitaxy, a large-area, homogeneous
5 heatability of the substrates.

It is customary to use hot-wall reactors without a rotating substrate for the deposition of SiC layers on current 2'' (inch) substrates.

10

These reactors have the drawback that the growth rate drops considerably over the path length of the gas. To compensate for this effect, according to the prior art the reactor height is varied over the path length, in
15 order to achieve homogeneous growth on the wafer.

A further drawback is the inhomogeneous growth perpendicular to the direction of flow, on account of the influence of the walls. Growth takes place at the
20 walls, and as a result additional process gases are consumed. Furthermore, the walls have an adverse effect on the flow profile in the reactor. In this case, according to the prior art an improvement is possible either by varying the process pressure and/or the flow
25 or by increasing the distance between the wafer and the wall.

A further drawback is the inhomogeneity of the doping over the path length and perpendicular to the path
30 length. In this case, primarily temperature inhomogeneities are of decisive importance, and such inhomogeneities, in the case of a hot-wall reactor without rotation, can only be improved by substantial outlay on equipment.

35

EP-A-0 164 928 has disclosed a vertical hot-wall reactor in which the substrates are stacked on top of one another. This requires a mechanical drive.

A mechanical drive for rotating the substrate(s) requires a mechanical passage to the hot substrate holder. This has the drawback that the passage leads to an inhomogeneity of the temperature of the substrate holder, that the mechanical elements, such as for example gears, lead to abrasion at the temperatures of over 1400°C which are required, so that, firstly, particles are generated and, secondly, materials are released, leading to undesirable background doping in the layers which are deposited.

A further drawback is the coating which is applied to the graphite of the substrate holder or substrate carrier for gastight sealing of the graphite surface. According to the prior art, SiC is used as this coating. The use of SiC has the drawback that the SiC coating, at the temperatures of over 1400°C required for the process, is etched by reactive hydrogen radicals, and therefore only a short service life of the graphite parts is ensured. Furthermore, the back surface of the substrate can be undesirably coated with SiC from the coated graphite surface as a result of close space epitaxy. The transfer of material results in holes forming in the SiC coating. Moreover, impurities are released through pores and holes in the SiC coating of the graphites, which impurities are incorporated in an electrically active manner, as foreign atoms, in the semiconductor layer and may influence the electrical properties of the layer. At the high process temperatures, hydrocarbons are released through pores and holes in the SiC coating of the graphites, which hydrocarbons increase the proportion of carbon in the vapor phase for the SiC epitaxy and therefore impair the controllability of the process.

In addition, in connection with the prior art, mention is made of the following documents, to which reference is expressly made moreover in connection with all the

conditions. In the form in which it is constructed as a radial flow reactor, the process gases flow from the center, through the temperature-controlled gas inlet, to the outside and over the rotated substrates into an exiting-gas collector at the outer periphery of the substrate holder or substrate carrier. The radial flow reactor advantageously does not have any walls, with the result that the above-described negative side wall effects of a hot flow channel reactor are avoided.

10

The rotation of the substrate plate(s) can advantageously be carried out by gas foil rotation, with the result that mechanical abrasion and complex mechanical bearing means and drives can be avoided.

15

The rotation of the substrate advantageously results in compensation of the dropping growth rate over the path length, and homogenization of any temperature gradients present in the substrate holder or substrate carrier perpendicular to the path length.

20

The rotation, and in particular the rotation by means of gas foil rotation, advantageously results in homogeneous growth in terms of layer thickness and doping and a homogeneous temperature distribution. Furthermore, it is firstly possible to achieve very low particle generation on account of the use of the gas foil rotation. Moreover, the fact that mechanical rotation at high temperatures without adverse effects on temperature homogeneity and service life of the components throws up problems which have not been solved to date is a factor in favor of gas foil rotation.

30

The boundary walls which face the process gas, in particular the substrate holder and the rotating substrate(s), can be heated to temperatures of up to 1800°C by means of high-frequency heating, lamp heating,

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resistance heating or any desired combination of such heating means. It is advantageously possible to use temperature control devices which heat the boundaries to identical or different temperatures. Consequently,
5 the process conditions can be varied or set very specifically.

For heating purposes, in particular with high-frequency heating, it is possible for one or more coils to be
10 disposed around or above and below the susceptor or substrate holder, in order to allow heat transfer to proceed optimally and with low losses and few control problems.

15 Separate control of the temperature of two or in each case two opposite boundary walls of the heated flow channel is also possible by using two separate heating circuits, each with a dedicated control means.

20 In particular, by controlling the temperature of the substrate-side boundary wall separately from the opposite boundary wall of the heated flow channel, through the use of two separate heating circuits each with a dedicated control means, it is advantageously
25 possible to fix the temperature gradient perpendicular to the substrate. In this way, the formation of Si clusters and seeds in the gas stream is advantageously reduced.

30 When two separately controlled heating circuits are being used for, respectively, the temperature of the substrate-side boundary wall and the temperature of the opposite boundary wall, it is therefore possible for the temperatures to be set separately. Consequently, it
35 is advantageously possible to set constant temperature gradients between the substrate and the opposite boundary wall of the flow channel.

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The boundary walls of the heated flow channel which face the process gas, and in particular the substrate carrier or substrate holder and the substrate plate(s), may in particular be constructed from highly conductive material, in order in this way also to enhance or improve the homogeneity of the materials produced.

Furthermore, the boundary walls of the heated flow channel which face the process gas, and in particular the substrate plates and substrate holders, are advantageously protected by a coating, e.g. TaC, which cannot be etched by hydrogen radicals, does not sublime at temperatures of up to 1800°C and is applied to the graphite of the substrate plates or substrate holders, in such a way that the surface of the coating remains continuous even at high temperatures and for long application times. The avoidance of free graphite surfaces therefore advantageously minimizes the release of impurities from the graphite. Consequently, undesired background doping can be limited to $< 5 \times 10^{14} \text{ cm}^{-3}$. The stable, gastight sealing advantageously suppresses the formation of additional hydrocarbons. The controllability of the composition of the vapor phase comprising silicon and carbon in the immediate vicinity of the substrate is thereby increased.

Close space epitaxy on the back surface of the substrate is advantageously avoided by the use of temperature-resistant coatings which are resistant to etching by hydrogen radicals. Such coatings for the substrate plate made from graphite may consist, in particular, of, for example, TaC.

The gases which enter the substrate holder or substrate carrier, which is constructed as a flow channel, are kept well below the decomposition temperature of the process gases until just before they enter, by means of

an actively cooled inlet. Therefore, the decomposition of the process gases is advantageously prevented as far as possible until just before the substrate. Consequently, vapor-phase deposition can only take
5 place for the first time above the substrate.

The cooled inlet and the hot substrate holder or substrate carrier are preferably only thermally separated and connected to one another by a narrow,
10 strongly thermally insulating segment. This is an advantageous thermal insulation measure, since it is simple.

Therefore, these two preceding measures allow high
15 efficiency of the process gases which are to be deposited, since they are only decomposed just before reaching the substrate. In addition, the abrupt temperature transition suppresses preferential depletion of a gas species in a temperature range,
20 which is used to make the composition of the vapor phase in the immediate vicinity of the substrate more controllable.

The low temperature gradient perpendicular to the
25 substrate holder or substrate carrier in the hot flow channel results in effective decomposition of the source gases. The abrupt temperature transition between gas inlet and substrate holder or substrate carrier and the low temperature gradient perpendicular to the
30 substrate holder or substrate carrier reduces the likelihood of the formation of Si clusters or seeds in the gas stream. This advantageously results in the growth rate being maximized.

35 Consequently, in a flow channel of this type with integrated substrate holder or substrate carrier, growth rates of $>10 \mu\text{m/h}$ are achieved.

The configuration of the flow channel downstream of the substrate holder or substrate carrier with outlet segments made from different inert materials avoids reactions of the gases flowing out, and in this way the homogeneity of the materials which are to be produced is also improved. The process conditions are therefore reproducible. Uncontrollable influences caused by reactions of the gases flowing out are avoided.

10 The introduction of thin bodies made from inert materials with different conductivities (e.g. Ta, Mo) from the substrate holder on or in the substrate holder advantageously serves to influence the temperature distribution in the latter, irrespective of the coil position.

In the other embodiment of the heated flow channel with rotating substrates, in the form of a radial flow reactor, it is moreover advantageous for the boundary wall of the heated flow channel which lies opposite the substrate to be rotatably connected to the substrate-side boundary of the heated flow channel at a specific distance therefrom. This results in an improved rotary movement of the at least one substrate within the flow channel in order to achieve optimum homogeneity of the semiconductor layers produced.

Furthermore, it is advantageous for the boundary wall of the heated flow channel which lies opposite the substrate to be actively coolable by a gaseous medium in order to provide the desired temperature gradient. In this way, it is possible to have an advantageous influence on the temperature gradients and the temperature/time profiles.

35 Since the rotating substrate can be positioned by means of a substrate holder disposed on any desired boundary wall of the heated flow channel, gravity effects can

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advantageously be used deliberately for process optimization.

If the gas outlet of the substrate holder is
 5 constructed as a gas distributor ring, the gases can be
 discharged from the flow channel uniformly over the
 periphery. Constructing the gas distributor ring from
 different inert materials leads to the temperature
 gradients and temperature/time profiles being
 10 advantageously influenced. In this case too, the number
 of process parameters which can be influenced is
 advantageously increased. Reactions of the emerging
 gases are thereby avoided.

15 The corresponding method according to the invention can
 advantageously be executed in such a way, by using
 corresponding process and carrier gases, by optimum
 temperature management in combination with suitable
 pressures, that layers are deposited very homogeneously
 20 and with high growth rates. It is intended, in
 particular, to improve known deposition methods, such
 as CVD, MOCVD or MOVPE methods.

The introduction of process and carrier gas which has
 25 been cooled to well below the process temperature just
 ahead of the hot substrate avoids premature
 decomposition of source gases and local supersaturation
 of the gas stream with a decomposition product.

30 Selected process and carrier gases which have an
 advantageous influence on the quality of the
 semiconductor layers produced are used in the method
 according to the invention.

35 In particular, doping of from $5 \times 10^{14} \text{ cm}^{-3}$ to $1 \times 10^{19} \text{ cm}^{-3}$
 is achieved.

The complete decomposition of source gases ahead of or above the substrate advantageously, on account of the homogeneous temperature profile of the substrate holder, also leads to growth rates of 10 $\mu\text{m/h}$ or more
5 for SiC and/or $\text{SiC}_x\text{Ge}_{1-x}$ ($x=0-1$) semiconductor layers.

Low temperature gradients perpendicular to the substrate which are produced using the method according to the invention advantageously lead to the reduction
10 of the formation of Si clusters and seeds in the gas stream.

Homoepitaxial or heteroepitaxial deposition is advantageously possible.
15

Brief Description of the Drawing

The invention is described below by way of example, without the general idea of the invention being
20 restricted, on the basis of exemplary embodiments and with reference to the drawing, to which, moreover, reference is expressly made in connection with the disclosure of all the inventive details which are not explained further in the text. In the drawings:

25

Fig. 1: shows an illustration of a device according to the invention for the deposition of layers from the vapor phase, in cross section,

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Fig. 2: shows an illustration of the temperature profile as a function of the location within the device according to fig.1,

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Fig. 3 shows an illustration of a further device according to the invention, configured as a radial flow reactor with dual rotation, for the deposition of layers from the vapor phase, in cross section,

The reference numeral 3a denotes a substrate holder or substrate carrier with a substrate plate 4 which rotates as a result of gas foil rotation, these components both comprising conductive material (e.g. graphite) which can withstand high temperatures, with a coating (e.g. TaC) which is inert and is able to withstand hydrogen radicals even at temperatures of up to 1800°C. Opposite the substrate holder 3a there is an upper boundary 3b of the flow channel, for the purpose of forming, with side walls (not shown), a flow channel which is closed off perpendicular to the gas flow direction and in which the substrate holder 3a is integrated. The substrate holder 3a may also be disposed on other boundary walls. The reference numeral 5 denotes one or more coils which are disposed around or above and below the closed substrate holder 3a, in order to actively heat the entire flow channel. The reference numeral 6 denotes one or more outlet segments made from different materials, in order to continuously reduce the temperature between susceptor and gas

outlet. The reference numeral 8 denotes plates, which are thin compared to the substrate holder 3a, of materials which have a different electrical conductivity from the substrate holder 3a and are inert (e.g. Mo, Te), in order to influence the temperature distribution independently of the coil position.

Fig. 2 shows the temperature profile within the system as a function of the location of the process gases in the system. It can be seen that the process gases are cooled all the way until they enter the heated flow channel, where they are then very rapidly brought to temperatures which are required for pyrolysis of the process gases. After the outlet, continuous and controlled cooling of the process gases is effected by means of the outlet segments.

Fig. 3 shows a further exemplary embodiment of a system according to the invention or a device according to the invention, which is formed as a radial flow reactor with dual rotation. The reactor is constructed symmetrically with respect to the dot-dashed line on the left-hand side of fig. 3, i.e. only one half is shown. In this figure too, reference numeral 1 denotes an actively cooled inlet. The reference numeral 2 denotes a short insulation segment made from highly insulating, temperature-resistant material (e.g. graphite foam) between cold inlet 1 and hot susceptor. The reference numeral 3a denotes a substrate holder or substrate carrier with a substrate plate 4 which rotates as a result of gas foil rotation, both components consisting of material which is conductive and withstands high temperatures (e.g. graphite), with an inert coating (e.g. TaC) which withstands hydrogen radicals even at temperatures up to 1800°C. Opposite the substrate holder 3a there is a boundary 3b in order, together with side walls (not shown), to form a flow channel which is closed off perpendicular to the gas

flow direction and in which the substrate holder 3a is integrated. The substrate holder 3a may also be disposed on other boundary walls. The reference numeral 5 denotes one or more coils which are disposed around or above and below the closed substrate holder 3a, in order to actively heat the entire substrate holder 3a. The reference numeral 6 denotes one or more outlet segments made from different materials, in order to continuously reduce the temperature between susceptor and gas outlet. The reference numeral 8 denotes plates, which are thin compared to the substrate holder or substrate carrier 3a, made from materials which have a different electrical conductivity from the substrate holder 3a and are inert (e.g. Mo, Ta), in order to influence the temperature distribution independently of the coil position.

Unlike the system shown in fig.1, the reference numeral 7 additionally denotes, in the construction as a radial flow reactor with dual rotation, an outlet ring which ensures uniform flow distribution over the periphery of the substrate holder.

Fig. 4 shows the temperature profile for the system in the construction as a radial flow reactor with dual rotation; this temperature profile in principle corresponds to the temperature profile according to fig.2.

PATENT CLAIMS

1. A method for depositing SiC and/or SiC_xGe_{1-x} (x=0-1) semiconductor layers or related materials with a wide (electronic) band gap and, in particular, a high binding energy (such as for example AlN, GaN) by means of a CVD method, characterized in that the at least one substrate is heated to a temperature of approx. 1100 to approx. 1800°C, in that the at least one substrate is rotated in a flow channel reactor which is actively heated on all sides, and in that the coating takes place homoepitaxially or heteroepitaxially.
2. The method according to claim 1, characterized by controlled heating of the walls of the flow channel reactor.
3. The method according to claim 1 or 2, characterized in that the rotation of the at least one substrate is effected by a mechanically driven shaft and/or preferably by "gas foil rotation".
4. The method according to claim 3, characterized in that the substrates are disposed on at least one substrate plate, which is disposed in or on a substrate holder, and in that the substrate plate(s) are driven relative to the substrate holder by "gas foil rotation".
5. The method according to claim 4, characterized in that a radial flow reactor with dual rotation is used as reactor.
6. The method according to one of claims 1 to 5, characterized in that the process and carrier gas(es) are introduced just ahead of the hot substrate, at a temperature which is well below

the process temperature, so that premature decomposition of process or source gases and/or local supersaturation of the gas stream with a decomposition product is avoided.

5

7. The method according to claim 6, characterized in that the gases are cooled before being introduced.

10

8. The method according to one of claims 1 to 7, characterized by the use of H_2 , N_2 , inert gases or mixtures thereof as carrier gas.

15

9. The method according to one of claims 1 to 8, characterized by the use of silane (SiH_4) or other Si-containing inorganic and organic starting materials, germane (GeH_4) and propane (C_3H_8) or other hydrocarbon gases as process gases.

20

10. The method according to one of claims 1 to 9, characterized in that doped layers of $5 \times 10^{14} \text{ cm}^{-3}$ to $1 \times 10^{19} \text{ cm}^{-3}$ are produced.

25

11. The method according to one of claims 1 to 10, characterized in that by the complete decomposition of source gases ahead of or above the substrate, on account of the homogeneous temperature profile of the substrate holder, so that growth rates of $10 \text{ } \mu\text{m/h}$ or more are achieved for SiC and/or SiC_xGe_{1-x} ($x=0-1$) semiconductor layers.

30

35

12. The method according to one of claims 1 to 11, characterized in that the reduction of Si cluster and seed formation in the gas stream is achieved by low temperature gradients perpendicular to the substrate.

13. The method according to one of claims 1 to 12, characterized in that the layers are deposited at process pressures of between 10-1000 mbar.
- 5 14. The method according to one of claims 1 to 13, characterized in that the substrates consist of the same material or of a different, suitable material.
- 10 15. A device for producing SiC semiconductor layers and related materials with a wide electronic band gap and a high binding energy by means of a vapor-phase application method, and in particular a CVD method, having
 - 15 - a reactor chamber, which has at least one gas inlet for the reaction gases,
 - a rotatable substrate holder, on which the substrate(s) are disposed horizontally (next to one another),
 - 20 - a gas outlet,
 - a heater device, which actively heats both the substrate surfaces which are to be coated, to temperatures of from 1100 to 1800°C, and the wall region of the reactor chamber which lies
 - 25 opposite these surfaces, in a controlled manner, to high temperatures.
16. The device according to claim 15, characterized in that the reactor chamber is constructed in
 - 30 rotationally symmetrical form and has a central gas inlet and a rotationally symmetrical gas outlet.
17. The device according to claim 15 or 16, characterized in that the boundary walls of the reactor chamber, which face the reactor space, and the substrate plate(s) and/or substrate holder(s) have a continuous, inert coating which is able to
- 35

withstand high temperatures of up to 1800°C and cannot be etched by hydrogen radicals.

18. The device according to claim 17, characterized in
5 that the coating consists of TaC, NbC, etc.
19. The device according to one of claims 15 to 18,
characterized in that the reactor is a radial flow
reactor with dual rotation which is, in
10 particular, of symmetrical construction.
20. The device according to one of claims 15 to 19,
characterized by a turning device for rotation of
the at least one substrate in each case on a
15 substrate plate which is disposed in or on a
substrate holder, by means of "gas foil rotation".
21. The device according to one of claims 15 to 20,
characterized by a turning device for rotation of
20 the at least one substrate in each case on a
substrate plate, which is disposed in or on a
substrate holder, by means of a mechanically
driven shaft.
22. The device according to one of claims 15 to 21,
characterized by at least one temperature control
device for providing a uniform temperature to all
25 the boundary walls facing the process gas, as top
side, underside and side walls of the heated flow
channel which is thereby closed off.
30
23. The device according to one of claims 15 to 22,
characterized by at least one temperature control
device for providing different temperatures at the
35 boundary walls facing the process gas, as top
side, underside and side walls of the heated flow
channel which is thereby closed off.

24. The device according to one of claims 15 to 23,
characterized in that one or more high-frequency
heating means are provided for the purpose of
heating the boundary walls which face the process
gas, and in particular the substrate holder.
25. The device as claimed on one of claims 15 to 24,
characterized in that one or more lamp heating
means are provided for the purpose of heating the
boundary walls which face the process gas, and in
particular the substrate holder.
26. The device according to one of claims 15 to 25,
characterized in that one or more resistance
heating means are provided for the purpose of
heating the boundary walls which face the process
gas, and in particular the substrate holder.
27. The device according to one of claims 15 to 26,
characterized in that any desired combination of
high-frequency, lamp and resistance heating means
are provided for the purpose of heating the
boundary walls which face the process gas, and in
particular the substrate holder.
28. The device according to one of claims 15 to 27,
characterized in that separate control of the
temperature of two or in each case two opposite
boundary walls of the heated flow channel is
effected by using two separate heating circuits,
each with a dedicated control means.
29. The device according to one of claims 15 to 27,
characterized in that separate control of the
temperature of the substrate-side boundary wall
from the opposite boundary wall of the heated flow
channel is effected by using two separate heating
circuits, each with a dedicated control means.

30. The device according to one of claims 15 to 29,
characterized in that the boundary walls, which
face the process gas, of the heated flow channel,
5 and in particular the substrate plate(s) and/or
the substrate holder, are made from a highly
conductive material, such as graphite.
31. The device according to one of claims 15 to 30,
10 characterized in that the boundary walls, which
face the process gas, of the heated flow channel,
and in particular the substrate plate(s) and/or
the substrate holder, has a continuous, inert
coating of, for example, TaC, NbC, etc., which is
15 able to withstand high temperatures (up to approx.
1800°C) and cannot be etched by hydrogen radicals.
32. The device according to one of claims 15 to 31,
20 characterized in that a cooling device actively
cools the gas inlet, up to just before the heated
flow channel, by means of a liquid or gaseous
medium.
33. The device according to one of claims 15 to 32,
25 characterized in that the cool gas inlet is sealed
with respect to the flow channel which is heated
on all sides by means of a highly insulating,
narrow adapter piece.
- 30 34. The device according to one of claims 15 to 33,
characterized in that the flow channel, downstream
of the actively heated zone, comprises outlet
segments which have different inert materials
(e.g. TaC-coated graphite, SiC-coated graphite,
35 quartz, etc.).
35. The device according to one of claims 15 to 34,
characterized in that thin plates, compared to the

thickness of the substrate holder, of inert materials (e.g. Ta, Mo, W) with a different electrical conductivity from the substrate holder can be fitted on or in the substrate holder, in order to locally influence the introduction of high frequency and therefore the input of energy.

36. The device according to one of claims 15 to 35, characterized in that the boundary wall, which lies opposite the substrate, of the heated flow channel is installed in a fixed position, at a defined distance from the substrate-side boundary of the heated flow channel, or is rotatably connected thereto.

37. The device according to one of claims 15 to 36, characterized in that the boundary wall, which lies opposite the substrate, of the heated flow channel can be actively cooled by a gaseous medium.

38. The device according to one of claims 15 to 37, characterized in that the rotating substrate can be positioned by means of a substrate holder disposed on any desired boundary wall of the heated flow channel.

39. The device according to one of claims 15 to 38, characterized in that the gas outlet of the substrate holder is formed as a gas distributor ring.

WO 01/14619 A1

Fig. 1

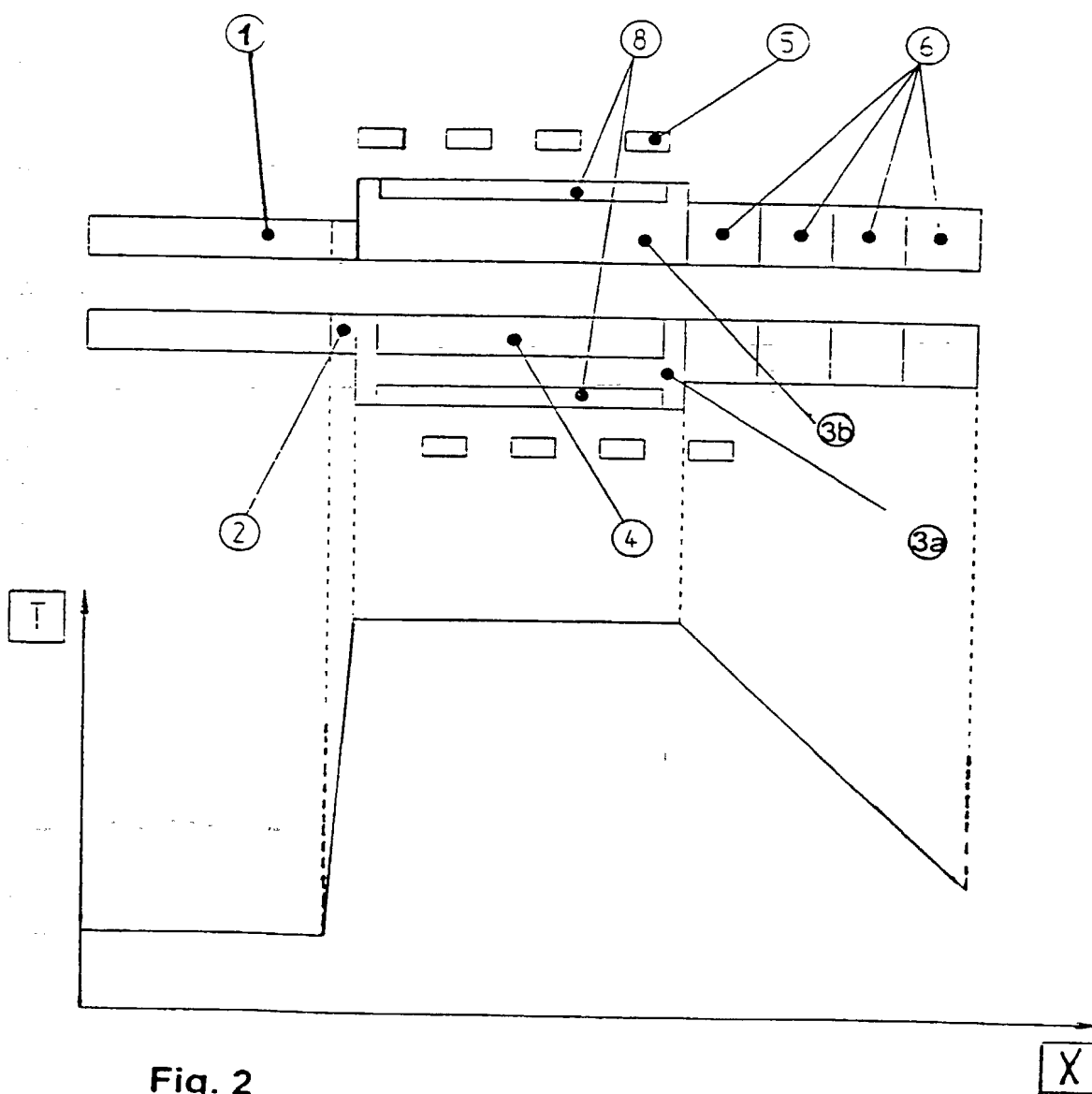


Fig. 2

Fig. 3

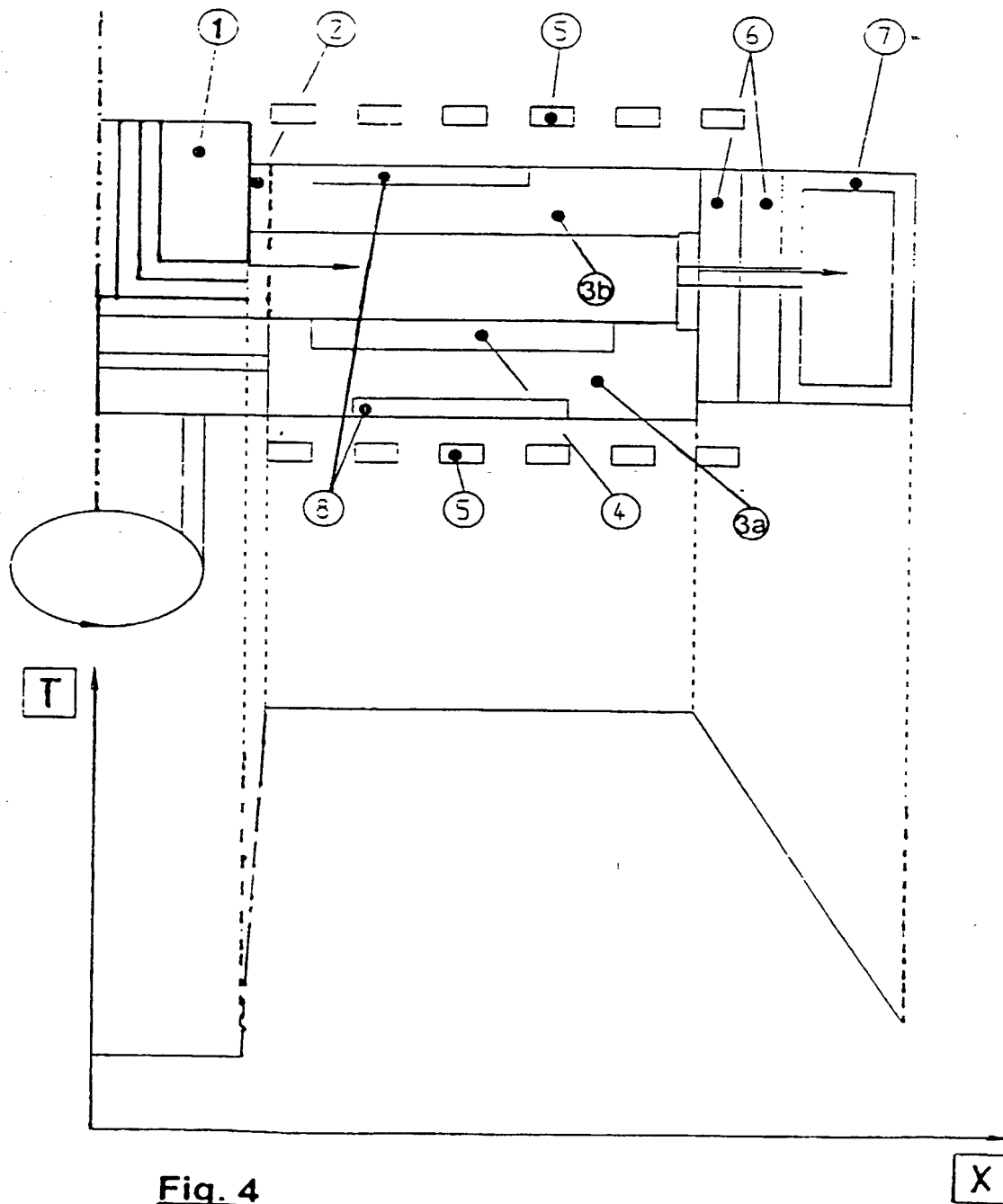


Fig. 4

OMBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY

(See Reference to PCT International Applications)

24230PCT/US

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

METHOD AND DEVICE FOR DEPOSITING MATERIALS WITH A WIDE ELECTRONIC BAND

GAP AND A HIGH BINDING ENERGY

the specification of which (check only one item below):

☐ is attached hereto.

☐ was filed as United States application

Serial No. _____

on _____

and was amended

on _____ (if applicable).

☒ was filed as PCT international application

Number PCT/DE00/02890

on August 24, 2000

and was amended under PCT Article 19

on _____ (if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations, §1.56(a).

I hereby claim foreign priority benefits under Title 35, United States Code, §119 of any foreign application(s) for patent or inventor's certificate or of any PCT international application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application(s) for patent or inventor's certificate or any PCT international application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed:

OR FOREIGN/PCT APPLICATION(S) AND ANY PRIORITY CLAIMS UNDER 35 U.S.C. 119:

| COUNTRY (If PCT indicate PCT) | APPLICATION NUMBER | DATE OF FILING (day month, year) | PRIORITY CLAIM UNDER 35 USC 119 |
|----------------------------------|--------------------|-------------------------------------|---|
| Germany | 199 40 033.4 | 24/08/1999 | <input checked="" type="checkbox"/> YES <input type="checkbox"/> NO |
| | | | <input type="checkbox"/> YES <input type="checkbox"/> NO |
| | | | <input type="checkbox"/> YES <input type="checkbox"/> NO |
| | | | <input type="checkbox"/> YES <input type="checkbox"/> NO |
| | | | <input type="checkbox"/> YES <input type="checkbox"/> NO |

Combined Declaration For Patent Application and Power of Attorney (Continued)
 (Includes Reference to PCT International Applications)

 ATTORNEY'S SUBJECT NUMBER
 24230PCT/US

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) or PCT international application(s) designating the United States of America that is/are listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in that/those prior application(s) in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, §1.56(a) which occurred between the filing date of the prior application(s) and the national or PCT international filing date of this application:

PRIOR U.S. APPLICATIONS OR PCT INTERNATIONAL APPLICATIONS DESIGNATING THE U.S. FOR BENEFIT UNDER 35 U.S.C. 120:

| U.S. APPLICATIONS | | STATUS (Check one) | | |
|---------------------------------------|------------------|---------------------------------------|---------|-----------|
| U.S. APPLICATION NUMBER | U.S. FILING DATE | PATENTED | PENDING | ABANDONED |
| | | | | |
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| PCT APPLICATIONS DESIGNATING THE U.S. | | | | |
| PCT APPLICATION NO. | PCT FILING DATE | U.S. SERIAL NUMBERS ASSIGNED (if any) | | |
| | | | | |
| | | | | |
| | | | | |

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith. (List name and registration number) MARTIN A. FARBER, Esq., Reg. No. 22,345
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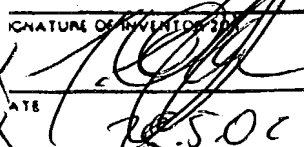
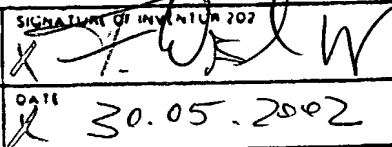
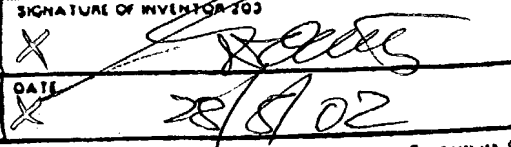
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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

| | | |
|--|---|---|
| SIGNATURE OF INVENTOR 1-00 | SIGNATURE OF INVENTOR 2-00 | SIGNATURE OF INVENTOR 3-00 |
|  |  |  |
| DATE | DATE | DATE |
| <u>28.5.02</u> | <u>30.05.2002</u> | <u>28/5/02</u> |

Combined Declaration For Patent Application and Power of Attorney (Continued)
 (Includes Reference to PCT International Applications)

 ATTORNEY'S SUCCESOR'S NAME
 24230PCT/US

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) or PCT international application(s) designating the United States of America that is/are listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in that/those prior application(s) in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, §1.56(a) which occurred between the filing date of the prior application(s) and the national or PCT international filing date of this application:

PRIOR U.S. APPLICATIONS OR PCT INTERNATIONAL APPLICATIONS DESIGNATING THE U.S. FOR BENEFIT UNDER 35 U.S.C. 120:
U.S. APPLICATIONS
STATUS (Check one)

U.S. APPLICATION NUMBER

U.S. FILING DATE

PATENTED

PENDING

ABANDONED

PCT APPLICATIONS DESIGNATING THE U.S.

PCT APPLICATION NO.

PCT FILING DATE

U.S. SERIAL NUMBERS
ASSIGNED (if any)

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith. (List name and registration number) **MARTIN A. FARBER, Esq., Reg. No. 22,345**
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| | POST OFFICE ADDRESS | POST OFFICE ADDRESS | CITY | STATE & ZIP CODE/COUNTRY |
| 202 | FULL NAME OF INVENTOR | FAMILY NAME | FIRST GIVEN NAME | SECOND GIVEN NAME |
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| | POST OFFICE ADDRESS | POST OFFICE ADDRESS | CITY | STATE & ZIP CODE/COUNTRY |
| 203 | FULL NAME OF INVENTOR | FAMILY NAME | FIRST GIVEN NAME | SECOND GIVEN NAME |
| | RESIDENCE & CITIZENSHIP | CITY | STATE OR FOREIGN COUNTRY | COUNTRY OF CITIZENSHIP |
| | POST OFFICE ADDRESS | POST OFFICE ADDRESS | CITY | STATE & ZIP CODE/COUNTRY |

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

| | | |
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| DATE | DATE | DATE |